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TECHNICAL MEMORANDUM 2022

MOISTURE SORPTION OF LEAD BETA RESORCYLATE SALTS

BY

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OCTOBER 1971

PICATINNY ARSENAL
DOVER, NEW JERSEY

15

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)* Picatinny Arsenal, Dover, New Jersey	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

MOISTURE SORPTION OF LEAD BETA RESORCYLATE SALTS

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

5. AUTHOR(S) *(First name, middle initial, last name)*

Daniel R. Satriana

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
October 1971	14	

8a. CONTRACT OR GRANT NO. b. PROJECT NO. DA 1M262[illegible]302A211 c. AMCMS Code 5222.11.58500 d.	9a. ORIGINATOR'S REPORT NUMBER(S) 2042 Technical Memorandum ~~[illegible]~~ 9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

10. DISTRIBUTION STATEMENT

Approved for public release: distribution unlimited.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY

13. ABSTRACT

Determination by differential thermal analysis and X-ray spectroscopy, of water sorption capabilities of lead beta resorcylate is described.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Lead Beta Resorcylate						
Dibasic Lead Beta Resorcylate						
Monobasic Lead Beta Resorcylate						
Beta Resorcylic Acid						

Security Classification

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DA Project: 1M262302A211
AMCMS Code: 5222.11.58500

Propellants Division
Feltman Research Laboratories
Picatinny Arsenal
Dover, New Jersey

ACKNOWLEDGEMENTS

Thanks are due to Mr. N. Gelber for the chemical determinations, Mr. J. Campisi for the X-ray diffraction data and Mr. E. Jones for the differential thermal analyses.

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ABSTRACT

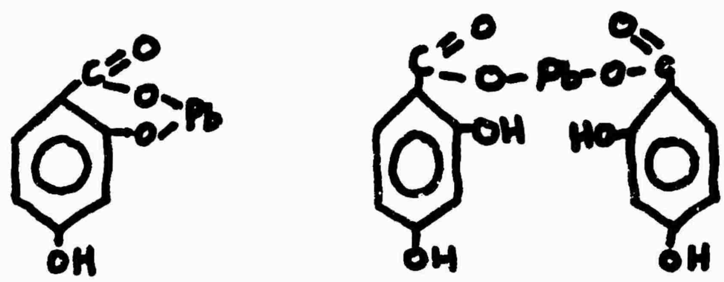
Determination by differential thermal analysis and X-ray spectroscopy, of water sorption capabilities of lead beta resorcylate is described.

CONCLUSIONS

Dibasic lead beta resorcylate (LBR) can be converted to a monohydrate on exposure to moisture. The hydrate has a unique crystal structure that can be distinguished from the anhydrous form by X-ray diffraction. Also, both forms are interconvertible without decomposition.

INTRODUCTION

Two LBR salts are known: the monobasic and the dibasic compounds. Their probable structural formulas are



Monobasic Dibasic

The method used commercially in their preparation has not been divulged. The commercial material varies between batches, containing impure mixtures of the lead salts. Consequently, it was our objective to synthesize reproducibly pure monobasic and dibasic LBRs. As a result of this investigation, optimum conditions for the preparation of the pure salts were developed[1].

Infrared spectroscopy was one of the techniques used to characterize the pure LBR salts prepared in the laboratory. Spectra of the monobasic and the dibasic salts are shown in Figure 1. Better resolution of the OH and CH stretching frequencies is discernible in the spectrum of the monobasic salt, while the dibasic salt has a broad band at the same 3500-3000 cm^{-1} frequency range. These findings suggested that the difference was probably due to the presence of adsorbed or combined water. In the light of this observation, moisture sorption of the lead salts was studied.

DISCUSSION OF RESULTS

Through the use of differential thermal analysis (DTA) it was found that the thermal characteristics of the two salts are different. It is evident from Figure 2 that the thermogram of the monobasic salt

1. Picatinny Arsenal Technical Memorandum 2021, dated October 1971.

has no endothermic transition, while the dibasic salt has two endothermic responses, one at 150°C., the other at 200°C. The endotherm appearing at 150°C. suggested either adsorbed moisture or a polymorphic transition.

By employing the following special technique, it was demonstrated that the endotherm at 150°C. was probably due to water of hydration: a sample of the dibasic salt was heated to 175°C. in the Differential Scanning Calorimeter (DSC), then cooled and placed in a humidity chamber for several hours. When the sample was heated again to 175°C. in the calorimeter, the endotherm reappeared. The sample was once more cooled to room temperature, and subsequently reheated to 175°C. This time the endotherm did not appear. However, when the sample was placed in the humidity chamber for several hours and again heated to 175°C., the endotherm reappeared. The results of this experiment are graphically shown in Figure 3.

Confirmation of the hydrate form of the dibasic lead salt was also obtained by X-ray diffraction. X-ray diffraction patterns were taken of a sample of the dibasic salt dried at 100°C. for 24 hours, and of the same sample after being in a humidity chamber at 90% RH for 72 hours. The spectra are shown in Figure 4. The changes in the major diffraction peaks signify differences in crystal structure. Evidently, these changes were due to the formation of the hydrate. The X-ray diffraction pattern of the monobasic lead salt is also included for purposes of comparison.

Thermogravimetric analysis (TGA) was used to determine the extent of hydration. The thermal decomposition of a sample of the dibasic salt is shown in Figure 5. The loss in weight was stepwise, the initial step corresponding to the endotherm at 150°C. of the DTA curve (Figure 2) and the second step to the endotherm at 200°C. It is evident that the loss in weight due to the decomposition of the hydrate is approximately 3.5%, which corresponds closely to the theoretical value (3.4%) for the monohydrate.

EXPERIMENTAL

X-Ray Diffraction

A Norelco wide angle difractometer utilizing CuK_{α} X-radiation was used to obtain X-ray diffraction patterns.

Differential Analysis

The 950 DuPont Gravimetric Analyzer and the Differential Scanning Cell for a 900 DuPont Differential Analyzer were used to measure thermal properties.

Infrared Analysis

Infrared spectra were obtained by means of the KBr pellet technique with a Perkin-Elmer, Model 621, Grating Infrared Spectrophotometer. The pellets, containing approximately 0.5% sample, were pressed to 0.7mm thickness.

Laboratory Preparation of Lead Beta Resorcylates

1. Monobasic Lead Beta Resorcylate

11.16g (0.05 moles) of lead monoxide (PbO) is added to 100 ml of absolute ethyl alcohol containing 16.96g (0.11 moles) of practical grade beta resorcylic acid. The mixture is heated at 60-70°C. for 1.5 hours with constant stirring. The product is filtered while still warm, washed twice with a small amount of warm (60°C.) absolute alcohol, and dried in the oven at 60°C. for several hours. The weight of the monobasic salt is 16.1g (theory 18.0g) or 89.5% yield based on the amount of PbO used in the reaction. Analysis: Found-Pb-57.29%, C-23.70%, H-0.18%; Calculated - Pb-57.66%, C-23.40%, H-1.12%.

2. Dibasic Lead Beta Resorcylate

11.16g (0.05 moles) of lead monoxide (PbO) is added to 175 ml of 50% (by volume) of ethyl alcohol containing 23.1g (0.15 moles) of practical grade beta resorcylic acid. The mixture is stirred at ambient temperature under a current of air for five hours. The solid is filtered, washed with ice cold 50% ethyl alcohol, and dried in the oven at 100°C. to constant weight. The yield of pure dibasic lead beta resorcylate is 23.8g (theory 25.7g) or 92.5% yield based on the amount of PbO used in the reaction. Analysis: Found - Pb-40.54%, C -32.85%, H-2.10%; Calculated - Pb-40.35%, C-32.75%, H-1.96%.

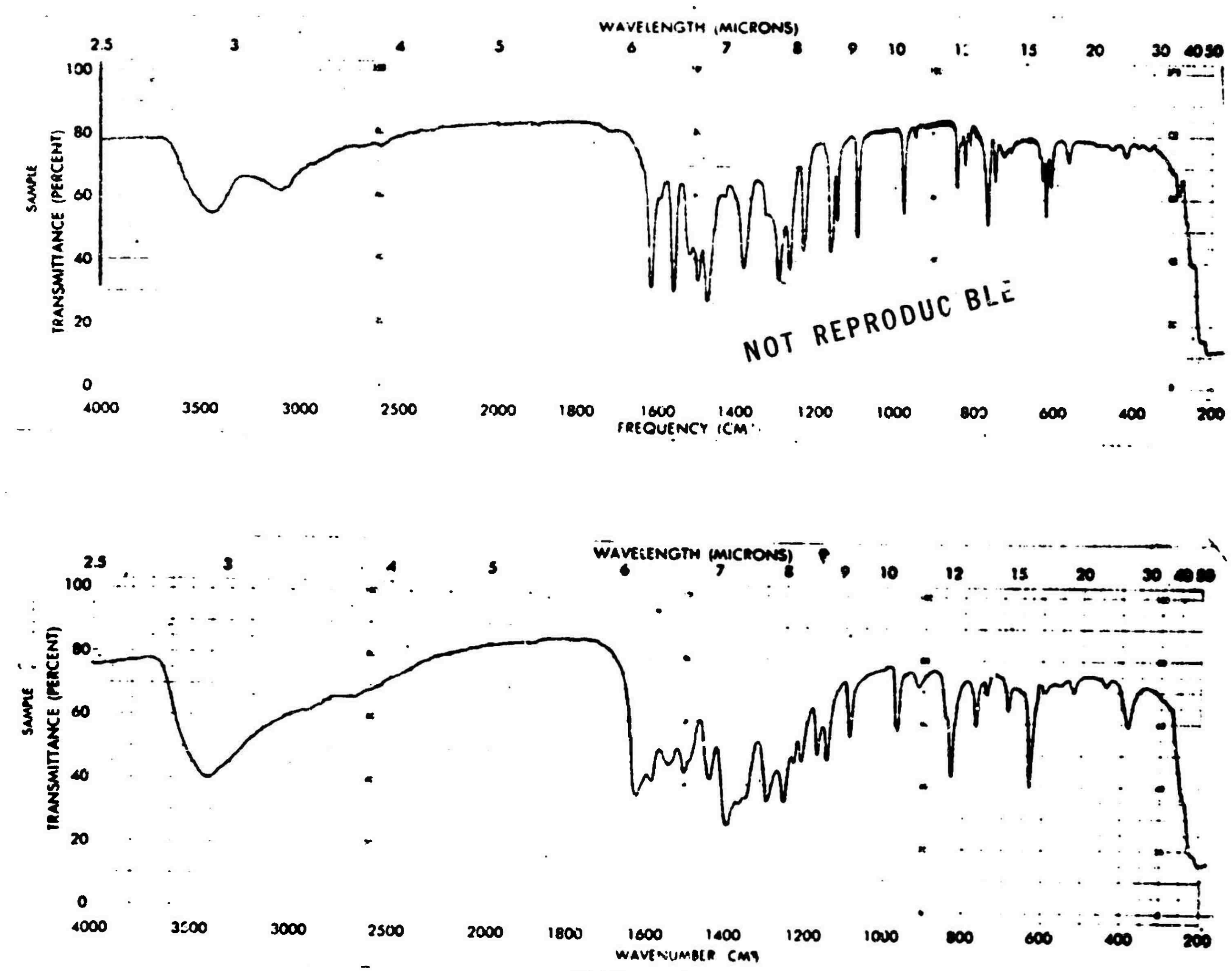


Fig. 1 Top - I.R. Spectra of Monobasic Lead Beta-Resorcylate
Bottom - I.R. Spectra of Dibasic Lead Beta-Resorcylate

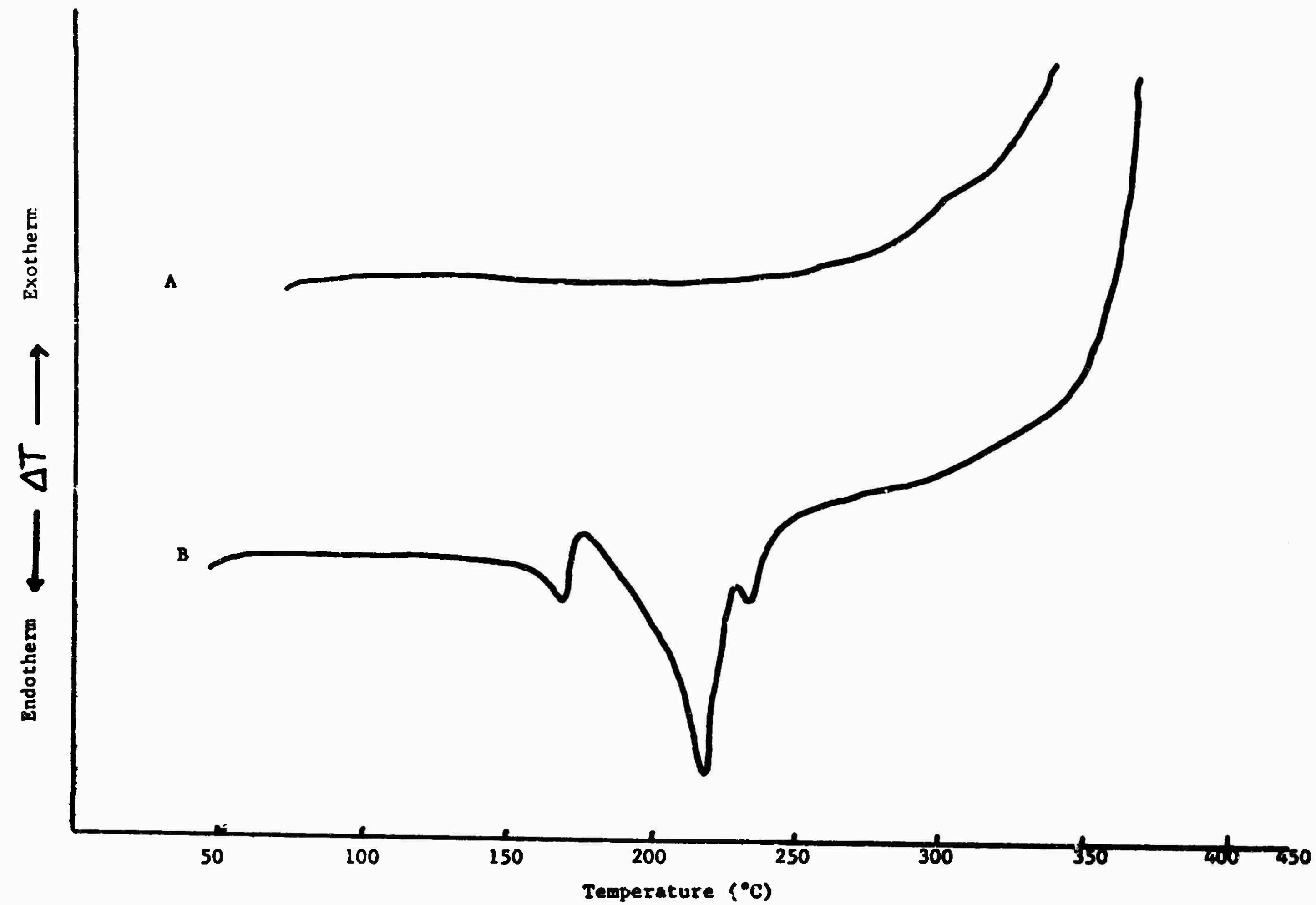


Fig. 2 A - Thermogram of Monobasic Lead Beta-Resorcylate
B - Thermogram of Dibasic Lead Beta-Resorcylate

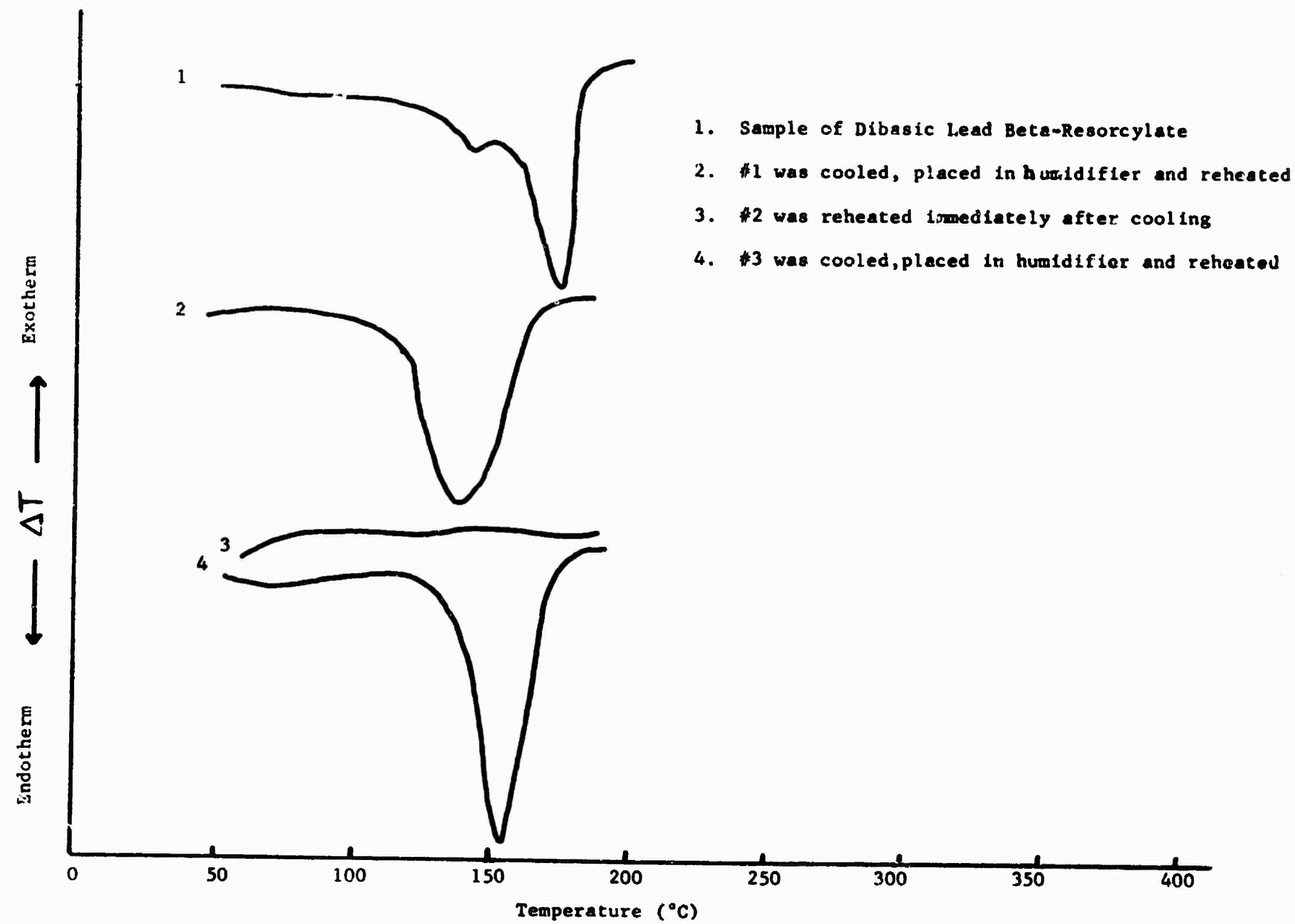


Fig. 3 Results of Experimental Treatment of Dibasic Lead Beta Resorcylate Salt In Differential Scanning Calorimeter

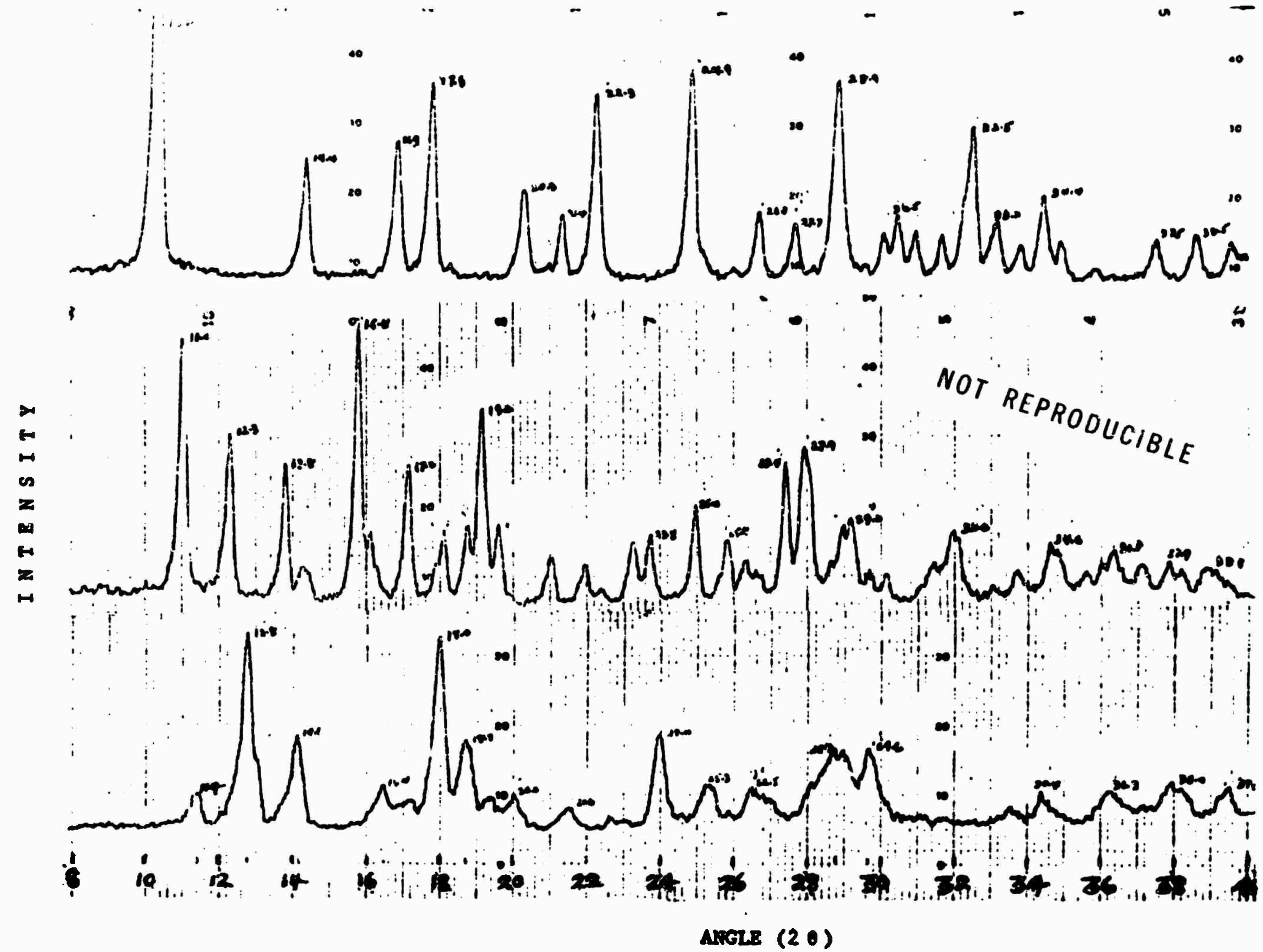


Fig. 4 Top - X-Ray diffraction pattern of monobasic lead beta-resorcylate
Middle - X-Ray diffraction pattern of dibasic lead beta-resorcylate (72 hrs at 90% RH)
Bottom - X-Ray diffraction pattern of dibasic lead beta-resorcylate (dried at 100°C, 24 hrs)

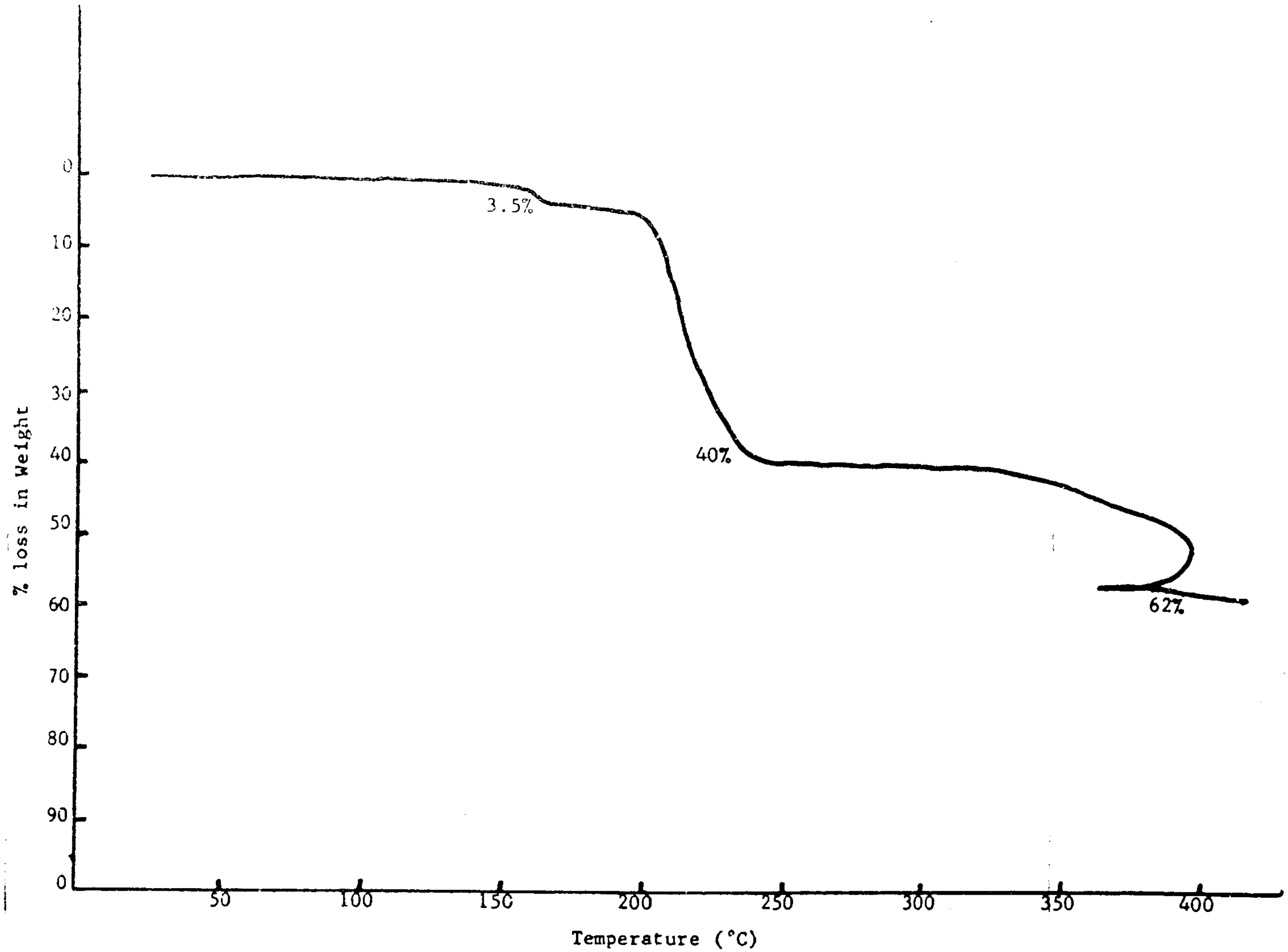


Fig. 5 Thermogravimetric Analysis of Dibasic Lead Beta-Resorcylate